

Argonne National Laboratory

AN AUTOGRAPHIC DILATOMETER
FOR USE WITH PYROPHORIC AND
ALPHA-ACTIVE MATERIALS

by

F. L. Yaggee and J. W. Styles

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9700 South Cass Avenue
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Metallurgy Division

December 1969

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AN AUTOGRAPHIC DILATOMETER FOR USE WITH PYROPHORIC AND ALPHA-ACTIVE MATERIALS

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ABSTRACT

A fused-quartz dilatometer was designed and constructed specifically for obtaining dilatation and phase-transformation data for plutonium-bearing alloys that are potential nuclear fuels. The apparatus can be installed and operated within a standard plutonium glovebox without sacrificing accuracy, sensitivity, or reproducibility in the experimental results. The dilatometer operates over the range from 25 to 1000°C, provides for programmed heating and cooling rates from 1/2 to 200°C/min, and will accommodate cylindrical, tubular, or sheet specimens that are from 1/4 to 2/5 in. in diameter (or width) and 1/4 to 2 in. in length. Dilatation runs can be made in vacuum (1×10^{-6} mm Hg) or in an inert gas environment of argon, helium, or nitrogen at normal glovebox pressures (≈ 740 mm Hg). A permanent record of the expansion versus temperature is automatically plotted. This equipment has a displacement (expansion) resolution of 2 μ in., an emf (temperature) resolution of 30 μ V, and an overall reproducibility of $\pm 0.1\%$. The dilatometer system can be calibrated with a Leeds and Northrup Type K-3 potentiometer.

Among the unique features incorporated in the apparatus design are (1) a mechanical means of verifying the electronic output representing specimen expansion, and (2) two matched electronic gaging heads for monitoring expansion displacement. The gaging heads may be used separately to monitor free expansion as a function of specimen temperature, or simultaneously to determine the expansion interaction between two dissimilar materials that are in intimate contact (coefficient of friction nearly unity) even when the materials exhibit widely different physical, mechanical, and thermal properties.

INTRODUCTION

Reliable thermal-expansion information about nuclear fuels and fuel-jacket alloys is imperative to the design, operation, and control of nuclear reactors. Considerations of reactor safety demand that the fuel exhibit a thermal-expansion behavior that is reproducible, positive, and moderate in magnitude, so that continuous control of core reactivity is facilitated. The fuel must also not undergo a solid-state phase transformation within

the operating temperature range. This latter condition is necessary to avoid dimensional distortion of the fuel geometry as a result of transformation-induced volume changes. All of these requirements, which qualify a potential fuel material as worthy of future investigation, can be readily checked by inspection of the analog record of expansion versus temperature.

Plutonium-bearing fuels, metallic (U-M, U-Pu-M)* or ceramic (UX, UX-PuX),** possess the greatest potential for applications in fast breeder reactors. Since these materials are strongly alpha active, highly toxic, and, for metallic fuels, dangerously pyrophoric, they must be handled in an inert atmosphere with complete containment at all times.^{1,2} The present dilatometer design is compatible with the space limitations of a glovebox installation without sacrificing accuracy, resolution, or reproducibility of the experimental data.

The thermal-expansion behavior of potential fuel-jacket alloys (i.e., austenitic stainless steel or vanadium-base alloys) is as important as that of the fuels because it directly affects the thermal stresses that are generated in the fuel jacket under normal conditions of reactor operation, and even more so during sudden reactor transients. Dilatation specimens of potential fuel-jacket alloys may be in the form of solid cylinders, rectangular sheets, or cylindrical tubes.

The difference between the thermal expansion of the fuel and the jacket is of special importance in studying the phenomenon of thermal ratcheting, which is observed in nuclear fuel elements under certain conditions of high fuel burnup. This phenomenon arises from mechanical interaction between the fuel and the jacket after intimate physical contact has been established. Thereafter, each component exerts a significant influence on the expansion behavior of the other, and neither this influence nor the observed response of the assembly can be inferred, by purely analytical means, from the individual data for free-expansion conditions. The restricted-expansion behavior of the fuel and jacket materials is not generally reproducible on successive heating and cooling cycles because of the plastic strain that occurs in the components and the subsequent change in the coefficient of friction at the contact surface. This dilatometer embodies a capability for obtaining reliable estimates of the stress developed at the interface between the fuel alloy and the jacket material.

DESCRIPTION OF EQUIPMENT

The dilatometer is shown schematically in Fig. 1 in a representative setup for obtaining dilatation data for a cylindrical specimen under free-expansion conditions. Dilatation runs are conducted in a vacuum environment

*M denotes a metallic constituent such as Mo, Zr, or Ti.

**V denotes a nonmetallic constituent such as C, S, or P.

within the bell jar and the fused-quartz tube that contains the specimen. The base of the apparatus is cooled by water to maintain a uniform temperature and to protect the vacuum seals. The equipment installation within the plutonium glovebox is shown in Fig. 2, and the associated temperature-programming and data-recording equipment is shown in Fig. 3.

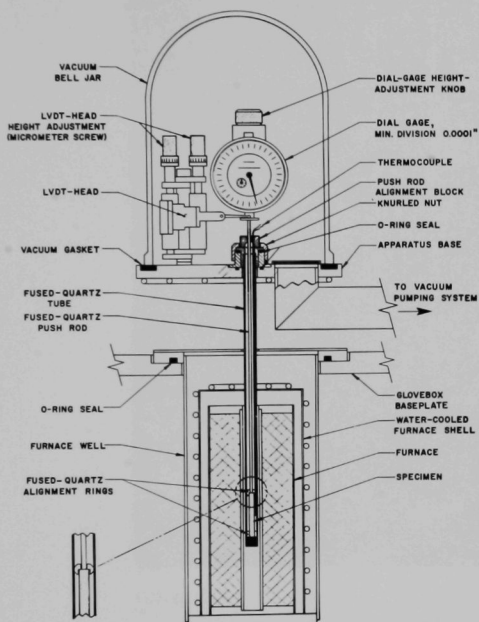


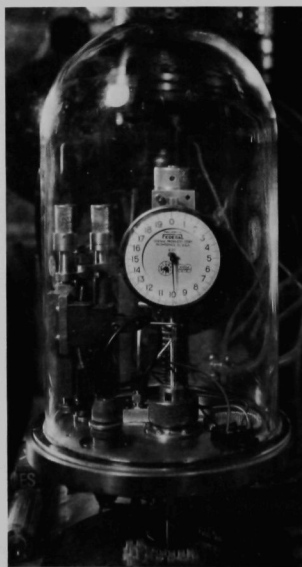
Fig. 1

Schematic of an Autographic
Fused-quartz Dilatometer

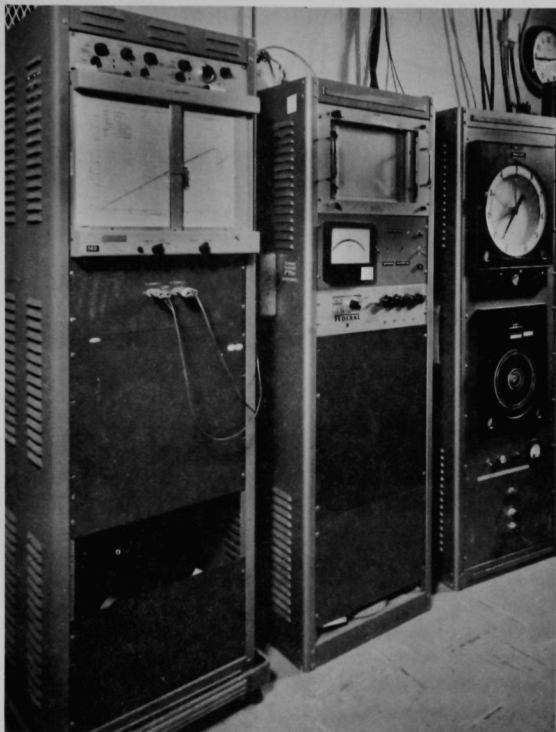
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Fig. 2

Dilatometer Installation in
a Plutonium Glovebox



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Fig. 3. Temperature-programming and Autographic-readout
Instrumentation for a Fused-quartz Dilatometer

The quartz specimen tube is sealed to a ground quartz plug at the bottom end and fitted with a brass collar at the upper end. An elastomer "O" ring (Viton A) provides the vacuum seal between the collar and the base of the dilatometer. Each collar is hand fitted to the individual specimen tube to obtain a very close-tolerance fit. The glass-to-metal seal between the frosted tube surface and the collar is usually made with an indium-tin solder, but a high-temperature wax can also be used. Both sealants have been used with equal success, provided that a very close fit (within 0.0002-0.0005 in.) existed between the collar and the specimen tube. Specimen tubes with diametral variations that exceed this tolerance are unsatisfactory. The specimen tube is held in position by a knurled slotted nut (see Figs. 1 and 2), which is secured after the dilatation experiment has been assembled with the push rod and thermocouple in place. Quartz alignment rings are used to center and guide the specimen and the push rod. These rings have a minimum contact area with the walls of the

specimen tube and are polished to reduce the sliding friction. Specimens with diameters (or widths) from 1/8 to 1/2 in. are accommodated by using specimen tubes of appropriate size.

The various push-rod arrangements used with this apparatus are illustrated in Fig. 4. A single push rod (a 0.12-in.-dia quartz tube) is used

to accommodate tube or sheet specimens under free-expansion conditions. A concentric arrangement of two push rods is used in conjunction with two linearly variable differential transformers (LVDT heads) to obtain data on the restricted expansion of either component (core or jacket) in a simulated fuel-element assembly. The outer push rod is a 0.24-in.-OD quartz tube; the top end is similar in appearance to the bottom end shown in Fig. 4. The top surface of the flat disk is the contact area for the displacement sensor of one of the LVDT heads. All quartz components (specimen tube, push rod, and alignment rings) are made of high-quality, bubble-free, fused quartz obtained from the same commercial source and traceable to the same production batch.

Free expansion of the specimen is monitored with the LVDT head, as shown schematically in Fig. 1. The actual size and shape of the LVDT head can be seen in Fig. 2. Two LVDT heads are used to monitor the relative expansion

between the fuel alloy and jacket material in a simulated fuel-jacket assembly (see Fig. 4). Expansion is simultaneously displayed on a mechanical dial gage that has a total displacement range of 0.200 in. and a minimum graduation of 0.0001 in. The dial gage is used primarily as a check on the LVDT head; both sensors should agree within 0.0001 in. In the event of a malfunction in the electronic measuring system, the dial gage can be used as an alternative, though less accurate, displacement indicator for the remainder of the dilatation run. The electronic measuring system (LVDT head and amplifier) produces an output voltage proportional to the displacement, and the output is linear over a 0.040-in.-displacement range. This

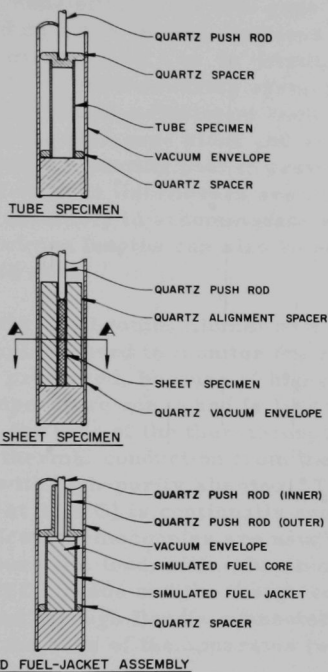


Fig. 4. Push-rod Arrangements Used to Accommodate Dilatation Specimens of Different Geometries

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output is continually recorded as the ordinate on an X-Y plotter. The two LVDT heads are mounted on separate micrometer screws, which provide accurate height adjustment at the beginning of a dilatation run. The vertically oriented holes in the micrometer screw assembly (see Fig. 2) provide the coarse height adjustment for the LVDT heads. These holes receive two dowel pins that form part of the LVDT mounting bracket. The dial-gage assembly, consisting of the dial gage and the block to which it is attached, is mounted on a 5/8-in.-dia stainless steel post on top of a die spring that is 1 in. in diameter by 3 in. in length. This assembly is converted into a rigid displacement-measuring system by compressing the die spring with the dial-gage height-adjustment knob. Two Vlier screws are threaded into the back of the dial-gage block and are seated in a vertical groove in the stainless steel mounting post to prevent rotation of the dial-gage assembly. Spacers of various thicknesses are inserted between the die spring and the dial-gage assembly to accommodate specimens of different lengths. Different specimen lengths can also be accommodated by using shorter or longer push rods.

Either a Chromel/Alumel or a platinum/platinum-10% rhodium thermocouple is used to monitor the specimen temperature. Although the former is preferred, because of higher output voltage, the latter has a higher temperature range and is less susceptible to oxidation. In both instances, the size of the thermocouple wire is small (0.010-in. diameter) to reduce thermal conduction from the specimen.* The thermocouple is insulated with high-purity alumina. The thermocouple output (relative to a junction at 31.4°C) is continually recorded on the abscissa of the X-Y plotter. New thermocouples are usually checked at the respective melting points of pure tin, lead, zinc, aluminum, and silver. The output leads from both the LVDT heads and the thermocouple are brought out of the vacuum environment through Bendix connectors and Conax seals, respectively, located in the base of the apparatus (see Fig. 2).

The dilatometer furnace is located in a well beneath the baseplate of the glovebox enclosure. The furnace is 4 in. in diameter and 12 in. in length, and operates in the same nitrogen environment as the rest of the glovebox system. A removable water-cooled shell surrounds the furnace to reduce the amount of heat dissipated to the well enclosure. The furnace core is noninductively wound on a recrystallized alumina tube of 1 1/4-in. OD (1-in. ID) and 12-in. length with 20-gage Kanthal A wire. The 9.5-in.-long winding is wound with 8.8 turns/in. over a distance of 1 in. from either end and 5.5 turns/in. over the remaining 7.5-in. length. Refractory cement is used to cover the windings. The core is mounted in a larger recrystallized-alumina tube (2 5/16-in. OD, 1 3/4-in. ID, and 12 in. long) to provide a 1/8-in. annulus (dead space) between the winding and the larger tube. This spacing is achieved by means of machined grooves in the lava end plates of the

* The thermocouple legs are spot-welded to opposite sides of the specimen.

furnace assembly. The uniform temperature zone within the furnace core is determined by the positions of two serrated cylindrical heat-reflecting shields (0.015-in.-thick platinum) that fit snugly around the outer alumina tube. The shields are oriented with the serrations pointing toward the center of the furnace winding, and are positioned by trial and error to achieve the most uniform temperature profile. Another dead space is provided between the platinum shields and the stainless steel outer shell of the furnace.

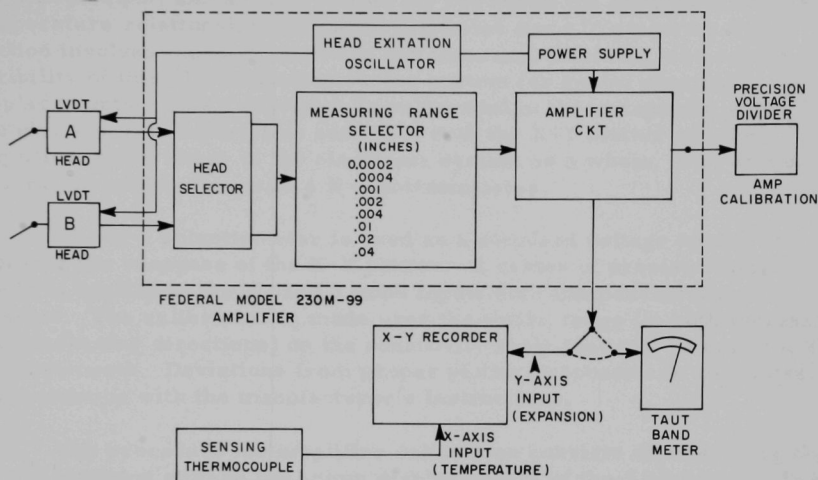
OPERATION OF EQUIPMENT

The specimen, push rod, thermocouple, and necessary alignment rings are assembled within the specimen tube with the latter removed from the apparatus. The dial-gage assembly must be rotated 90° to either side of its normal position (see Fig. 2) to provide the necessary clearance for removal and reinsertion of the specimen tube. After the specimen tube has been secured in position with the slotted nut, the dial-gage assembly is returned to its normal position. Spacers are then inserted, as needed, between the die spring and the dial-gage assembly to accommodate the specimen length without changing the push-rod length. The die spring is compressed with the dial-gage height-adjustment nut, and the Vlier screws are set in place, as described. An arbitrary initial dial-gage reading (usually between 0.010 and 0.020 in.) is used as the dial-gage reference.

The LVDT head is mounted on the vertical, micrometer screw assembly, as previously described, with the sensor extended, as shown in Fig. 2. The sensor is moved downward about its pivot point until the sensor tip makes contact with the flat button-head surface that is part of the dial indicator. A friction clutch at the pivot point permits the sensor to be rotated through 180° and used in any position within this arc. Temperature variations in the vicinity of the LVDT head will not result in displacement error when the head is used in the position shown in Fig. 2. After selecting the measuring range to be used in the dilatation run, final adjustment of the LVDT head is made with the micrometer adjustment screw. The proper measuring range selected is just greater than the anticipated expansion. The amplifier output is observed on the center-zero taut-band meter shown in the circuit in Fig. 5 when making the final LVDT head adjustment. A full meter displacement to the left of center indicates the beginning of the linear measuring range for any range. The apparatus is then evacuated, the amplifier output switched to the X-Y plotter, and the temperature programmer turned on to begin the dilatation run.

The specimen heating rate is controlled by means of a cam-type temperature programmer by using 12-in.-dia cams. These cams are accurately machined from either sheet metal or Lucite, and provide heating rates between 1/2 to 200°C/min. For specimens with very low thermal conductivity,

the cam programmer is interrupted at temperatures between 25 and 100°C, and the specimen is allowed to equilibrate thermally for 30-40 min at each temperature step. Above 100°C a programmed heating and cooling rate of 1/2°C or larger is used, depending upon the specimen material. A seven-day timer is used to shut off the furnace power at the conclusion of a single run or series of runs, as desired.



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Fig. 5. Block Diagram of the Electronic Displacement-measuring System

The basic setup for making a dilatation run with a simulated jacketed fuel assembly is similar to that described for obtaining free-expansion data, except that two LVDT heads are used in conjunction with the concentric push-rod arrangement shown in Fig. 4. To obtain data on the restricted expansion behavior of either the core or the jacket, the appropriate LVDT head is given a positive polarity by means of a switch at the amplifier, and the second head is switched out of the circuit.

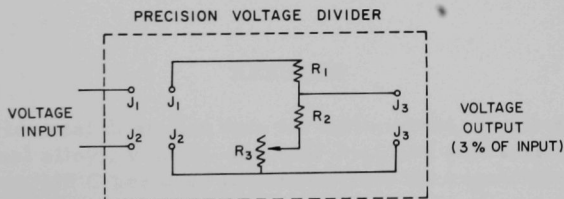
To obtain data about the relative expansion of the components of the jacketed assembly, both LVDT heads must be used with opposite polarities. The LVDT head monitoring the component that exhibits the higher expansivity (usually the reactor fuel) is assigned a positive polarity, and the second LVDT head a negative polarity. The resulting positive amplifier output represents the difference in expansion between the core and jacket, as affected by each other. The analog curve that is produced represents the difference in expansion as a function of temperature.

CALIBRATION

The electronic displacement-measuring system is shown schematically in Fig. 5. The system consists of the LVDT heads, an electronic amplifier with eight different ranges that correspond to expansions of from 0.0004 to 0.080 in. ($\pm 0.0002 - \pm 0.040$ in., respectively) at maximum amplifier output, and an X-Y plotter for recording the expansion versus temperature relationship during the dilatation run. Dilatometer calibration involves the verification of the accuracy, linearity, and reproducibility of this electronic measuring system for known physical displacements. This calibration is performed in three separate steps, namely, separate calibration checks of both the X-Y plotter and the amplifier, and a check of the electronic system as a whole. The entire calibration can be done with a K-3 potentiometer.

The K-3 potentiometer is used as a standard voltage source in checking the response of the X-Y plotter. A series of precise voltage levels is applied to the X- and Y-axis inputs both independently and together. The calibration is made over the entire range (in both increasing and decreasing directions) on the sensitivity scale that will be used for the measurements. Deviations from proper plotter response are corrected in accordance with the manufacturer's instructions.

The procedure for amplifier calibration consists of measuring the amplifier output voltage for known displacements of the LVDT head. In the calibration, the K-3 potentiometer is used as a precision voltmeter. A voltage divider (see Fig. 6) is required to reduce the amplifier output



R_1 - 1.1 MEG Ω PRECISION TOL. 1 %

R_2 - 32 K Ω PRECISION TOL. 1 %

R_3 - 5 K Ω TEN TURN PRECISION POTENTIOMETER

RESISTANCE TOL. 3 %

LINEARITY TOL. .05 %

J₁ } OFFSET BANANA JACKS - SHORTING PLUG REMOVED
J₂ } TO ISOLATE AMPLIFIER OUTPUT FROM
DIVIDER NETWORK.

J₃ - BANANA JACKS FOR READOUT OF REDUCED AMPL. OUTPUT.

voltage (± 50 V max) to the range of the K-3 potentiometer (1.5 V max). The voltage divider is adjusted by means of the precision potentiometer R3 to yield 3.0% of the input voltage. The LVDT head is displaced by known equal increments on either side of the midpoint of the linear measuring range, and the amplifier output (reduced by the divider) is recorded. The displacement standard is a calibrating fixture equipped with a differential micrometer screw that has a total travel of 0.050 in. and a readability of 0.000005 in. The calibration procedure is repeated a minimum of two times for increasing and decreasing displacements on each of two amplifier ranges. The first calibration run is made on the range just lower (high magnification) than that to be used in the dilatation run, and it is repeated on the actual range to be used. The positive polarity of the voltage-divider output is maintained during the calibration procedure by means of the polarity switch on the K-3 potentiometer. An inaccurate response is corrected by repeated adjustment of two multiturn precision resistors located in the amplifier circuit.

The final calibration check is made on the LVDT head, amplifier, and X-Y plotter, as a system, by observing the pen response for known displacements of the sensor arm of the LVDT head.

Since the two LVDT heads are matched, the same amplifier calibration should be applicable to both. This is easily checked by placing both LVDT heads in the calibration fixture and repeating the calibration procedure with the polarity switch for one head placed in the positive position and the other in the negative position. Under these conditions the amplifier output should be zero for all displacements of the differential micrometer spindle.

RESULTS

Experimental dilatation data for Armco iron, 63.3U-22.2Pu-14.5Zr* (a potential fuel alloy), V-20Ti, and AISI Type 316 stainless steel at temperatures up to 950°C are used to demonstrate the capabilities of this dilatometer. Both the Armco iron and the U-Pu-Zr alloy exhibit solid-state phase transformations (between 900-910°C and 598-660°C, respectively). These two materials are used to illustrate the form in which the experimental dilatation data are obtained and the response of the dilatometer to phase transformations. The V-20Ti alloy and the Type 316 stainless steel are used to demonstrate the phenomenon of restricted expansion exhibited by two dissimilar metals when they are thermally cycled while in intimate contact. Table I lists the data for the linear expansion of the four materials under free-expansion conditions. The overall accuracy of the dilatation results is within $\pm 2\%$.

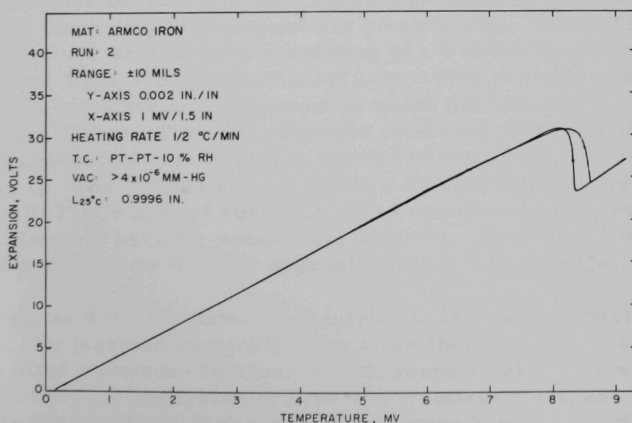
*Alloy compositions given in weight percent.

TABLE I. Linear Thermal Expansion of Selected Metals and Alloys

Composition of Material ^a	$\frac{L}{L_0^\circ\text{C}} = 1 + AT + BT^2$		Temperature Range, °C	Mean Expansion Coefficient, $10^{-6}/^\circ\text{C}$	Expansion, %
	A, 10^{-6}	B, 10^{-9}			
Armco Iron (alpha phase)	13.1	2.51	0-200	13.60	0.27
			0-400	14.10	0.56
			0-600	14.60	0.87
			0-800	15.10	1.20
			0-900	15.35	1.38
63.3U-22.2Pu-14.5Zr	10.4	12.8	0-200	12.96	0.25
			0-400	15.52	0.62
			0-500	15.80	0.84
			0-598	18.02	1.07
V-20Ti	8.78	1.87	0-200	9.15	0.18
			0-400	9.52	0.38
			0-600	9.90	0.59
			0-800	10.27	0.82
			0-960	10.65	1.06
316 SS	16.4	1.6	0-200	16.72	0.33
			0-400	17.04	0.68
			0-600	17.36	1.04
			0-800	17.68	1.41
			0-960	18.00	1.80

^aCompositions in weight percent.

The curve of expansion versus temperature for Armco iron is shown in Fig. 7. Expansion is plotted as the ordinate in volts of amplifier output; temperature is plotted on the abscissa in millivolts of thermocouple output. The thermocouple is platinum/platinum-10% rhodium. On slow heating the

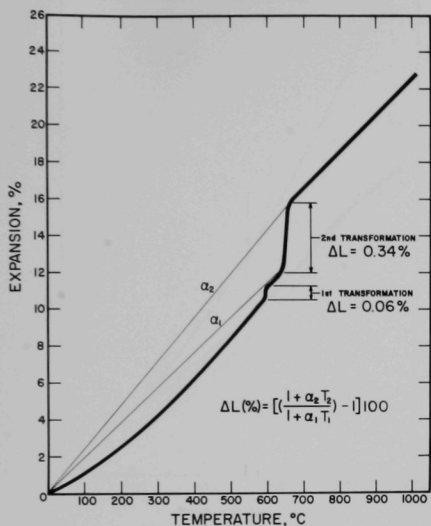


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Fig. 7. Typical Analog Record of Expansion versus Temperature for Armco Iron

$\alpha \rightarrow \gamma$ transformation in Armco iron occurs at 910°C , whereas on slow cooling the $\gamma \rightarrow \alpha$ transformation occurs at 902°C .³ The hysteresis in the transformation of Armco iron is illustrated in Fig. 7; the $\alpha \rightarrow \gamma$ transformation on heating occurs at 8.366 mV. The decrease in specimen length accompanying the

$\alpha \rightarrow \gamma$ transformation was 0.3%. Figure 7 also shows that, on cooling, the contraction curve of the alpha iron retraces its expansion curve.



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Fig. 8. Thermal-expansion Curve for the Potential Nuclear Fuel Alloy 63.3U-22.2Pu-14.5Zr (composition in weight percent)

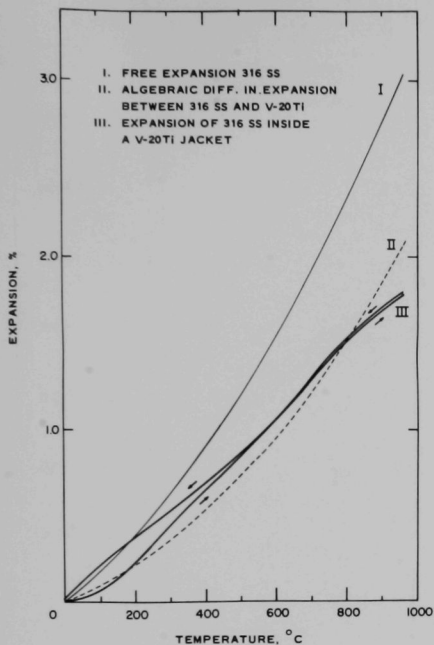
The 63.3U-22.2Pu-14.5Zr alloy exhibits two solid-state transformations between 25 and 950°C , as shown in Fig. 8. The first occurs at 595°C and is accompanied by a 0.06% increase in specimen length; the second occurs at 660°C , with a 0.34% increase in length. The total increase in specimen length on heating from 595 to 660°C is 0.52%. Above 660°C the fractional expansion of this alloy is given by

$$\frac{L}{L_{660^{\circ}\text{C}}} = 0.9871 + 1.941 \times 10^{-5} T, \quad (1)$$

where temperature T is in degrees centigrade.

Examples of the restricted-expansion behavior that may accompany the thermal ratcheting phenomenon are given in Figs. 9-12. These data were obtained for an assembly consisting of a V-20Ti tube that had been shrunk onto a Type 316 stainless steel core. This assembly represents an attempt to simulate a fuel element in which the fuel alloy has physically contacted the jacket. Type 316 stainless steel was chosen as the core material because its expansivity is similar to that of the U-Pu-Zr alloy (see Table I). The V-20Ti alloy exhibits a thermal expansivity representative of the V-Ti-Cr alloys⁴ currently under consideration as potential jacket materials for fast breeder reactor applications. The V-20Ti jacket of the simulated assembly is initially approximately 0.010 in. longer than the core.

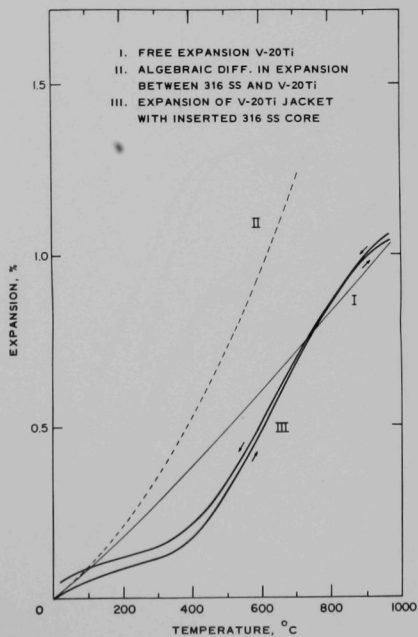
Figures 9-12 illustrate the restricted-expansion behavior of the core, the jacket, the jacketed assembly after a few thermal cycles, and the jacketed assembly after numerous thermal cycles, respectively. Curve I in Fig. 9 represents the free expansion of Type 316 stainless steel, and curve II represents the algebraic difference in expansion between the V-20Ti jacket and the core, as calculated from free-expansion data for the two materials.



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Fig. 10

Linear Thermal Expansion of a V-20Ti Jacket as Influenced by a Type 316 Stainless Steel Core in a Simulated Fuel-element Assembly.



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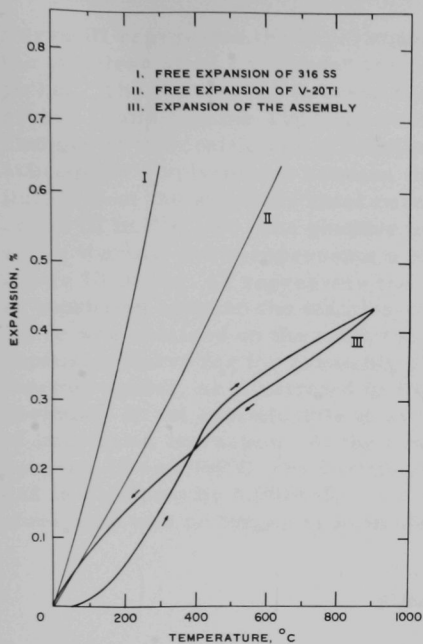


Fig. 11

Expansion Behavior of a Simulated Fuel-element Assembly after Six Thermal Cycles between 25 and 950°C.

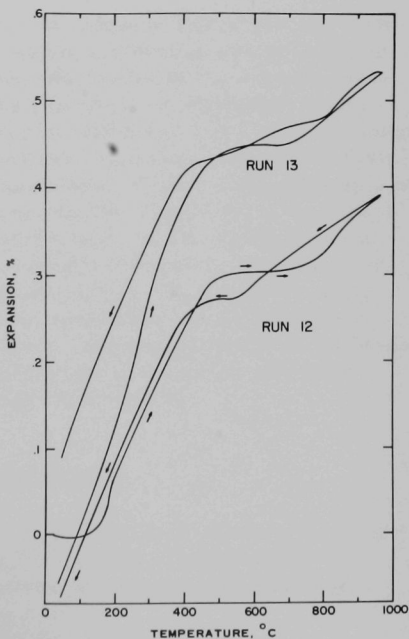


Fig. 12

Expansion Behavior of a Simulated Fuel-element Assembly after Numerous Thermal Cycles between 25 and 950°C.

Curve III represents the experimentally determined expansion behavior of the stainless steel core under the restrictive influence of the surrounding jacket. The differences between the free- and restricted-expansion curves (I and III), for Type 316 stainless steel in Fig. 9, are the result of changes in the coefficient of friction between the core and the jacket and subsequent displacement between these two components. The restrictive influence of the stainless steel core on the V-20Ti jacket is illustrated by curve III in Fig. 10. The positive expansion that remains at the conclusion of the thermal cycle represents a net plastic strain in the V-20Ti jacket. Curve III in Fig. 11 represents the experimentally determined difference in expansion between the stainless steel core and the V-20Ti jacket. This curve was obtained on the sixth thermal cycle between 25 and 950°C. The expansion curve for the assembly shows marked changes in subsequent thermal cycles, as illustrated in Fig. 12. The general shape of the curve obviously is not reproducible in successive thermal cycles, and the number of inflections increases. At the conclusion of the sixteenth thermal cycle between 25 and 950°C, the V-20Ti jacket had increased in length by 0.014 in. and in diameter by 0.0018 in., as a result of plastic strain. The stainless steel core was no longer in intimate contact with the jacket.

SUMMARY

The dilatometer design presented is uniquely suited for use with pyrophoric and alpha-active materials within a glovebox system without sacrificing accuracy, sensitivity, or reproducibility in the experimental results. It will accommodate cylindrical, tubular, or sheet specimens that vary from 1/4 to 2/5 in. in diameter or width and 1/4 to 2 in. in length. This equipment can be used to determine the expansion behavior of fuels and cladding materials (ΔL versus temperature), to detect the presence of solid-state transformations and to determine the magnitude of the length change associated with each such transformation, and to determine the restricted expansion behavior of a two-component system consisting of two dissimilar metals in intimate physical contact. In the latter application, information can be obtained on the restricted expansion behavior of either component or the entire assembly. Such information is indispensable to the study of the thermal ratcheting phenomena that are often observed in reactor fuel elements at high fuel burnups.

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